X-Ray Microtomography

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Introduction

Reconstructing a two dimensional function from its line integrals taken from many different directions is the basic process of tomography. When an x-ray beam is incident upon a specimen several related processes occur. If the incident x-ray beam completely penetrates the specimen and if the photons scattered or re-emitted from the interaction can also penetrate the entire specimen and if refraction effects are negligible, then a cross-sectional image of the interaction can be readily reconstructed. Tomographic experiments imaging these interactions fall into two broad categories, bright field, also called in-line techniques and dark field or off-axis techniques. In both techniques, the specimen is rotated to provide the different sampling directions. In a bright field, the specimen is placed directly between the x-ray source and detector. Spatial resolution is obtained by using either a small rastered probe beam or a position sensitive detector. Absorption and phase contrast are examples of bright field imaging. In dark field, a detector is placed adjacent to the specimen and inclined with respect to the x-ray illumination axis. A probe beam illuminates the specimen and the collected signal represents the line integral of the detected interaction along the linear path of the probe beam through the specimen. The attenuation of the probe beam is also monitored since dark field data must often be corrected using an absorption tomogram. Diffraction and fluorescence contrast are examples of dark field techniques. The use of position sensitive detectors in bright field imaging provides significant speed advantages over serially sampled dark field methods.

Perhaps the most widely recognized implementation of tomography is medical x-ray Computed Axial Tomography, referred to as a CAT or CT scan. In this imaging technique projections of the two dimensional cross-section of x-ray attenuation are acquired from many directions by placing the patient between a conventional x-ray source and a linear array detector. The source-detector pair is rotated about the patient to collect the projections from many angles. Reconstruction can be accomplished by a variety of algebraic, statistical of Fourier techniques. The geometry of projection, and the quality and completeness of the projection data determine the choice of technique. A Fourier technique called filtered back projection is the method commonly used in medical imaging. In both medical and industrial practice, the ability to image the internal structure of a macroscopic object without cutting provides clear advantages both to diagnosticians and patients and to quality control of fabricated parts.

X-ray absorption microtomography, referred to as XMT or μCT, is the extension of CAT scanning to specimens between 1mm and 1cm in size and creates cross sectional images with spatial resolution approaching 1 micron. Although the fundamental CT measurement remains the same for both large and small objects, the geometry of illumination, x-ray energy range and intensity requirements for small objects are well met by a synchrotron x-ray source. Perhaps most importantly, the tunability of a synchrotron source can be used to create tomographic images above and below the absorption edge of many elements and provides the ability to make three-dimensional images of the concentration of those elements.

Instrumentation

Beamline X2-B is designed to measure and reconstruct x-ray absorption. X2-B is a white beamline with a single crystal monochromator placed in the hutch. This simple design, built in 1990, was selected to provide an approximately 1cm wide by 5mm high illuminated area and to minimize sensitivity to vibration and source drift. Si<111> and flat multilayer mirrors with 2d = 22 and 44Å are available and can provide useful x-ray flux between ~7 to ~40 keV. The Si<111> is recommended for routine use and required for absorption edge crossing. The multilayers provide a nearly 100 fold increase in incident flux and are capable not only of shortening acquisition time but also of damaging the detector if not handled properly. Use of the multilayers must be approved by the PRT prior to experiments. Beamline motors for selecting x-ray energy, crystal position and tilt, and beam height are controlled using the data acquisition computer.

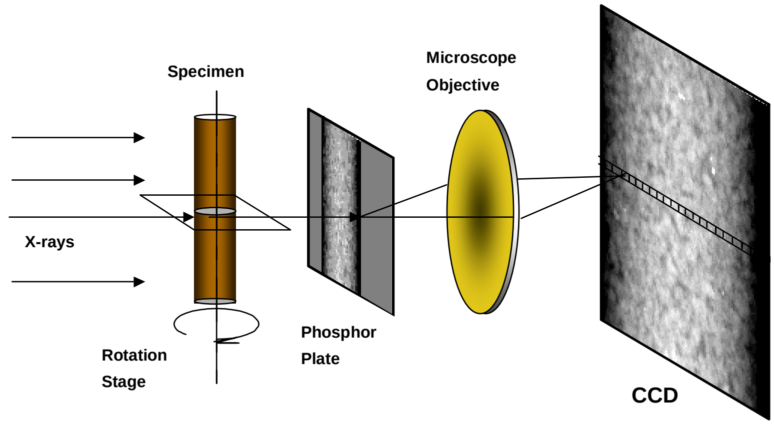

Figure 1

The geometry of illumination permits the use of a 2D position sensitive detector placed directly behind the specimen to simultaneously collect the projection data for many rays through the specimen. A schematic of the detector is shown in **Figure 1**. The detector consists of:

1. Motorized specimen micropositioners controlling specimen height, horizontal translation transverse to the x-ray beam and specimen rotation.
2. A single crystal scintillator to convert the pattern of x-ray intensity transmitted by the specimen to a visible light image. A 1cm square by 200micron thick polished CsI(Tl) crystal is optically isotropic, provides high yield, good stopping power relative to most specimens, good spatial resolution, and light output well matched to the CCD sensitivity. CsI(Tl) provides high quantum efficiency, however thin polished crystals are not commercially available. The crystal is attached to a focussing stage and is moved to refocus the image after a lens change. Care must be taken not to collide the scintillator into the specimen while refocussing.
3. A microscope objective magnifies the image onto the surface of a CCD. Image magnification is set by the objective selection. 2.5x, 5x, 10x or 20x Zeiss infinity corrected plan apochromats are provided. A 200mm tube lens gives a tube factor of 1.25x.
4. A thermoelectrically cooled Roper Scientific (Photometrics) CCD collects image data. The CCD is a back thinned 1024x1024 full frame device with 24micron pixels, ~340K electron full-well capacity, 14bit digitization, 800kHz readout, 16e/sec dark current at -35C.

Attenuation images are collected from many angles by rotating the specimen through small, evenly spaced, angular increments between 0 and 180 degrees using a rotation stage. The number of images needed is about $N\pi/2$ where N is the width of the specimen image in CCD pixels. A computer is used to control both the beamline configuration and the microtomography apparatus. The control software will be discussed in the data collection section.

A second computer performs the reconstruction process. Each collected 2D projection is analogous to a chest x-ray in medical imaging but is essentially free of the penumbral broadening and geometric distortion associated with source size and distance to the specimen. As indicated in **Figure 1**, the CCD is best thought of as a stack of linear detectors each associated with a single slice through the specimen. The distortion free projections make possible the reconstruction of perfectly registered 2D slices resulting in a 3D image. The parallel illumination also permits the use of a computationally efficient reconstruction technique called direct Fourier inversion that provides significant speed advantages in reconstructing the 1024 slices of a typical experiment.

Figure 2 shows the interior of the X2-B hutch. The monochromator crystal in the helium purged, lead lined tank to the left of the image. The gantry attached the two theta arm of the monochromator is levitated on air pads during energy changes. The beam propagates through about one meter of air before it is incident on the specimen and detector located to the right side of the image and shown in **Figure 3**. The specimen in this example is a carbon fiber reinforced

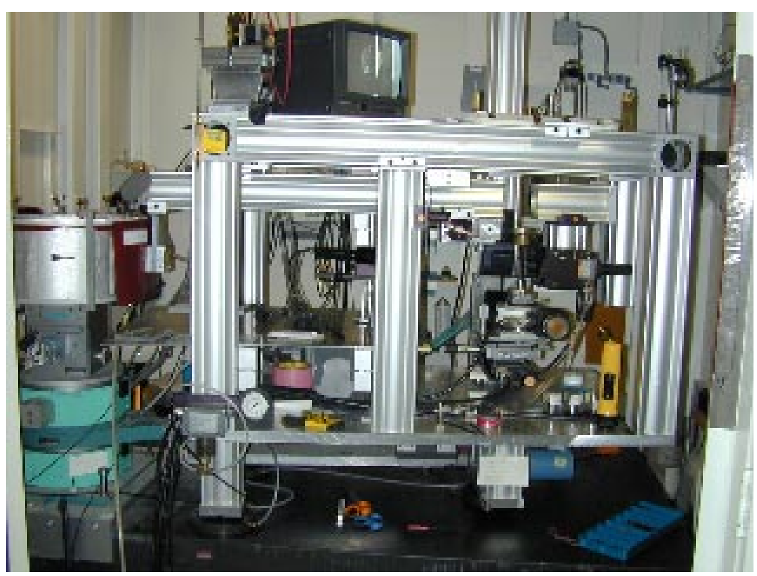

Figure 2

PEEK pressure vessel used to study gas evolution during depressurization in an oil saturated sandstone and illustrates the relatively large space available for specimen holders. Directly behind the specimen is the scintillator and focussing stage. The microscope objective, tube lens and turning mirror are internal and not visible.

Sample Preparation

Sample selection presumes the presence within the material of heterogeneity that will give rise to detectable absorption contrast. The specimen must also be dimensionally stable and able to withstand the high x-ray dose deposited. Specimen heating by the x-ray beam is usually insignificant. The mechanics of sample preparation are trivial and usually involve simply slicing off an appropriately sized piece of material for placement on the specimen rotation stage. Specimens are usually adhered using tacky wax to a post inserted in a eucentric goniometer. Epoxy tends to creep and may compromise specimen stability.

Specimen size selection is driven by straightforward but sometime conflicting criteria involving absorption, contrast and resolution. The specimen should absorb about 90% of the incident radiation along the most radio-opaque path to obtain the best signal to noise in the reconstructed image. The absorption of x-rays in the 7-40keV energy region covered by X2-B is described by

$$I/Io = e^{-\mu(\lambda)\rho x}$$

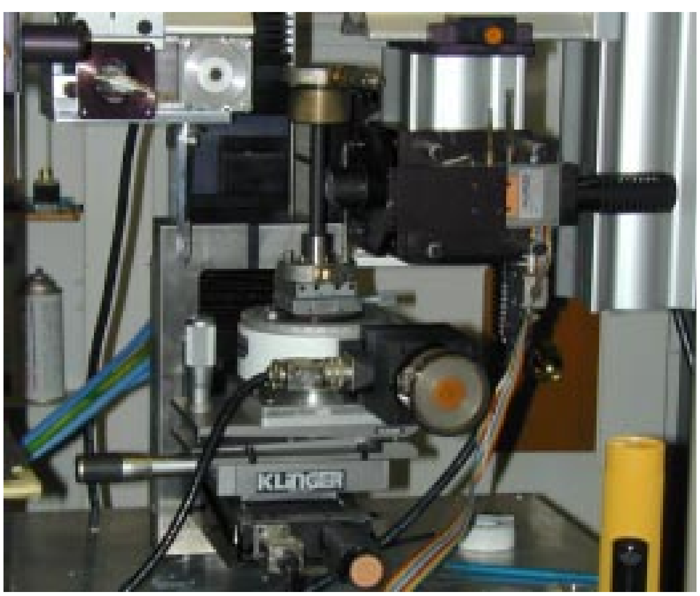

Figure 3

where I is the intensity of the absorbed x-ray beam, I_0 is the intensity of the incident x-ray beam, ρ is the specimen density, x is the specimen thickness, and

$$\mu(\lambda) = KZ^m\lambda^n$$

is the mass attenuation coefficient of the specimen where Z is the atomic number, m is approximately 4, λ is the x-ray wavelength and n may vary between 2.5 and 3. To absorb 90% of the incident radiation the quantity $\mu(\lambda)\rho x$, commonly referred to as τ should be approximately 2. To obtain $\tau\sim2$ the user can vary the specimen thickness and/or the x-ray energy. An increase in image noise is readily evident in images reconstructed from projection data where τ is less than 0.5. The detector response becomes non-linear when τ is greater than 2.5 and causes reconstruction artifacts. The strong atomic number and energy dependence provide ample contrast but makes the imaging of bulk samples of average atomic number greater than 20 difficult within the energy range available at X2-B. Similarly, low average atomic number specimens such as polymers and biological materials usually require the addition of a dopant to one or more of the components to provide sufficient absorption contrast.

As mentioned previously, magnification is selected by the choice of microscope objective. Ideally, the entire specimen should remain within the field of view of the CCD as it is rotated during a tomography experiment. Clearly, as the magnification is increased the field of view decreases and the specimen dimensions must be adjusted accordingly. For some specimens, the size required by the resolution criteria does not permit $\tau\sim2$ within the energy range available at X2-B. In other cases $\tau\sim2$ can only be obtained at x-ray energies above 20keV resulting in some resolution loss due to the finite stopping power of the scintillator.

If the specimen width remains completely within the field of view during CT then the reconstruction will be a quantitative map of linear attenuation coefficient. If the specimen or parts of the specimen container extend beyond the field of view, reconstruction is still possible using local techniques, however the resulting images will have a relative grayscale in arbitrary units.

The selection of specimen size, x-ray energy and resolution can also be driven by the differences in x-ray absorption among the different components of the specimen. For example, a specimen where resolution requirements are modest and mass absorption is low may indicate using a relatively large specimen size and high incident x-ray energy. If the contrast among the components of the sample is low then a lower x-ray energy and smaller sample size will probably be needed.

If a difference tomogram is to be acquired across an absorption edge then the x-ray energy and therefore specimen size is fixed. The lower limit of determination in a 3D image is about 1000ppm for many elements provided that $\tau \sim 2$ at the absorption edge energy. If a difference tomogram is to be acquired for more than one element then the size selection is a compromise depending on the concentration, edge positions and jump ratios of the elements of interest. The reconstruction software requires that projections to be subtracted for difference tomography must be in the same format.

Selection of the appropriate specimen size usually involves some a-priori knowledge of the specimen composition and structure. Spreadsheets for calculating the mass attenuation coefficient of compounds and the attenuation of samples of given size are available at the beamline. For true unknowns, a series of radiography experiments is needed to determine the appropriate specimen size based on its x-ray attenuation. Although there is considerable latitude in selecting specimen size, finding the optimum imaging conditions may require several adjustments.

Lastly, the design of specimen holders such as environmental or mechanical cells is the responsibility of the user who must take into consideration the x-ray absorption properties and geometry of the cell materials. Although a relatively large area is available for specimens, best absorption imaging is obtained when the specimen is in close proximity to the CsI scintillator.

Data Collection

The user controls the function of the microtomography system through the data acquisition computer running a PRT extended version of IPLab™, a commercial 2D image processing program. The PRT extensions to IPLab that provide motion control and image acquisition are available from the "Ext." menu. The details of the acquisition software are discussed in documentation available at the beamline and are presented during user training. The examples shown below are for information only and may differ slightly from those currently in use at X-2B. An example dialog box for simple image acquisition is shown in **Figure 4**. The popup Expose menu provides several kinds of exposure and alignment procedures. Similar dialogs are available for controlling the beamline motors, changing the x-ray energy, running a tomography experiment, sampling the tomography data and interactive reconstruction of single or small groups of slices.

For example, as discussed previously, initial radiography is needed to determine the specimen x-ray attenuation if the sample is of unknown composition and structure. This is done by first determining the exposure time for the incident x-ray beam at the initial estimated energy and then collecting a TauMap of the specimen using the menu option. TauMap collects radiographic images with and without the sample in the beam and calculates the log of I/Io. The specimen is moved during the TauMap process and a field is provided in the dialog box for the user to enter how far to move the specimen. Using this dialog the user can determine an exposure time, magnification, x-ray energy and sample size appropriate for the specimen.

Prior to a CT scan the specimen should be manually aligned on the eucentric goniometer to minimize specimen wobble about the rotation axis. Also the rotation stage axis must be aligned so that it is exactly parallel to the CCD columns and perpendicular to the incident x-ray beam. Automations such as *Find Axis, ScanAxis* and *Align Check* assist the user in evaluating the degree of misalignment. The user enters corrections using the beamline and CT stage motors. Alignments are discussed further during beamline training.

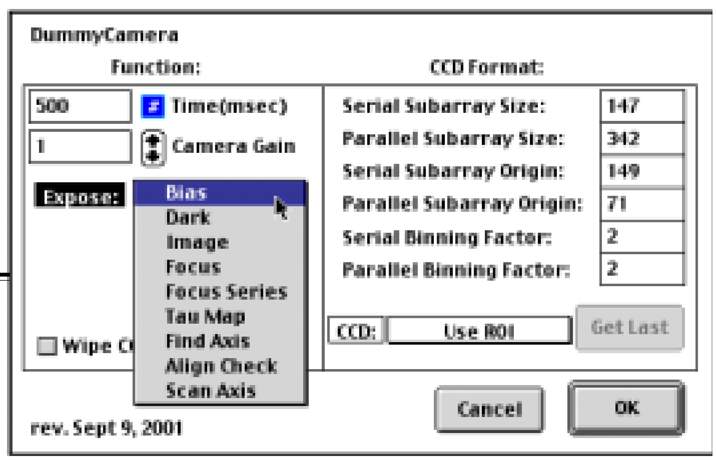

Figure 4

X2-B supports several CT scanning modes:

1. CCD binning and ROI acquisition, useful for quick low-resolution scans.

2. Global scanning, provides quantitative images of linear attenuation coefficient when the entire width of the specimen remains within the field of view during rotation.

3. Local scanning, provides qualitative grayscale images when all or part of the specimen extends beyond the field of view during rotation. Specimens should be no more than 3x the detector field of view.

4. Field doubling, the axis of rotation is placed by the user at either the right or left edge of the field of view and the specimen is rotated between 0 and 360 degrees. Field doubling collects 2048 wide data from the 1024 wide CCD.

5. Beam shaping for both global and local scans, the incident intensity profile is adjusted to provide nearly constant counting across the width of the specimen image.

6. Correlated sampling, improves S/N and removes most systematic errors from low contrast specimens. The specimen exposure time can be decreased by a factor of 2 with only a slight increase in noise compared to normal sampling.

The basic CT scanning protocol is automated. A scan begins by translating the specimen out of the x-ray beam and summing several Io beam images into a calibration buffer. The specimen is placed back into the beam and images are acquired at small angular increments between 0 and 180 degrees. Since the synchrotron ring output decays with time, the user may elect to repeat the calibration procedure periodically during the scan. The acquisition program computes 6000* ln(I/Io) for each specimen image and concatenates the result as a 16bit integer to a single file on

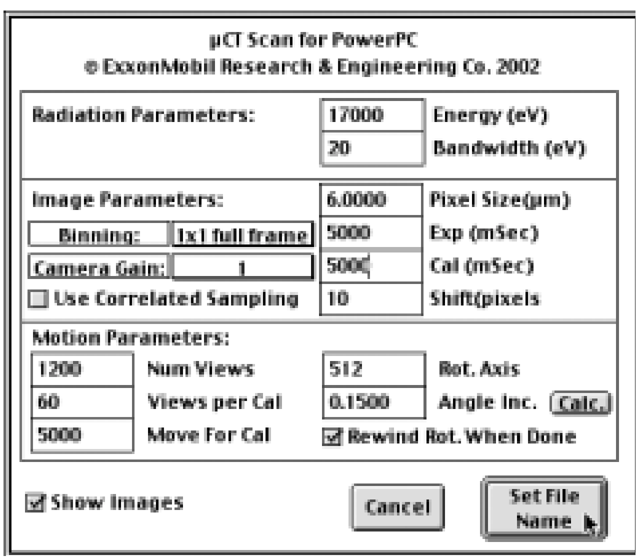

Figure 5

disk. Tomographic acquisition is initiated by the dialog box shown in **Figure 5**.

Local scanning and beam shaping are supported by providing separate exposure times for calibration and exposure. The maximum allowable exposure time for images of specimens that extend beyond the field of view of the CCD can be considerably longer than that used for the unattenuated beam since all points in the image are attenuated by the sample. Similarly, a beam shaper is available to match the pattern of incident intensity to the roughly cylindrical specimen shape allowing nearly constant count statistics across the field of view. It is important to collect as many counts in a single calibration and image frame as the CCD dynamic range will allow. These counting statistics have direct impact on the signal to noise of the reconstructed slices.

Scan times can vary considerably depending on the flux available from the synchrotron at a particular x-ray energy, the image magnification, the number of view angles needed, and the signal to noise required in the reconstructed volume. A low magnification 256 cube of data at about 20keV can be acquired in a few minutes. A single 1024 cube of data may need from 1 to 7 hours. The longest protocol supported will acquire a 2048x2048x1024 data volume using correlated sampling to reduce noise and usually requires the use of a multilayer mirror to provide sufficient flux to complete during a single x-ray ring fill.

An externally mounted SCSI or FireWire drive provided by the user and attached to the reconstruction computer is recommended since the data sets are sufficiently large to preclude practical transmission over current networks. Data transmission between the acquisition and reconstruction computers is asynchronous and causes no loss in acquisition speed for exposure times greater than 2 seconds.

IPLab provides a scripting utility that allows the user to construct extended or complex CT scanning procedures. For example, scripts that execute a series of CT scans and specimen height changes can be used either to scan multiple specimens or to create 3D volumes extended in the specimen height direction. Alternately, beam energy can be changed for mapping certain elements. The beamline is capable of extended unattended operation using IPLab scripts, however the instrument does not currently monitor the incident beam intensity.

Data Analysis

Once data Acquisition is complete, tomographic reconstruction can begin. Projection data is stored as a series of 2D images of x-ray opacity. If played back as a movie the projections simply show an image of a rotating specimen. The projections from a single row of pixels in the CCD must be collected together to form the sinogram data set necessary to reconstruct the corresponding 2D slice. A read function is available in IPLab to read projection data, reformat it into a sinogram and display it on the screen. The sinogram can then be reconstructed by selecting the parameters in the dialog box shown in **Figure 6**. As shown, the sinogram will be reconstructed with a rotation axis at column 128 without any adjustments or filtering using a global direct Fourier algorithm. The dialog contains options for conditioning the data to correct common artifacts and for region of interest reconstruction.

A free-standing application, CT_Recon, uses the parameters determined interactively in IPLab to reconstruct the entire 3D volume. The main dialog for CT_Recon requests the same information as shown in figure 6 and has additional fields for selecting the output format and range of slices. CT_recon supports drag-and-drop, import of tomography data in other formats, batch processing of multiple projection files and several output formats.

The final product of the reconstruction process is a single file containing a sequence of contiguous CT slices

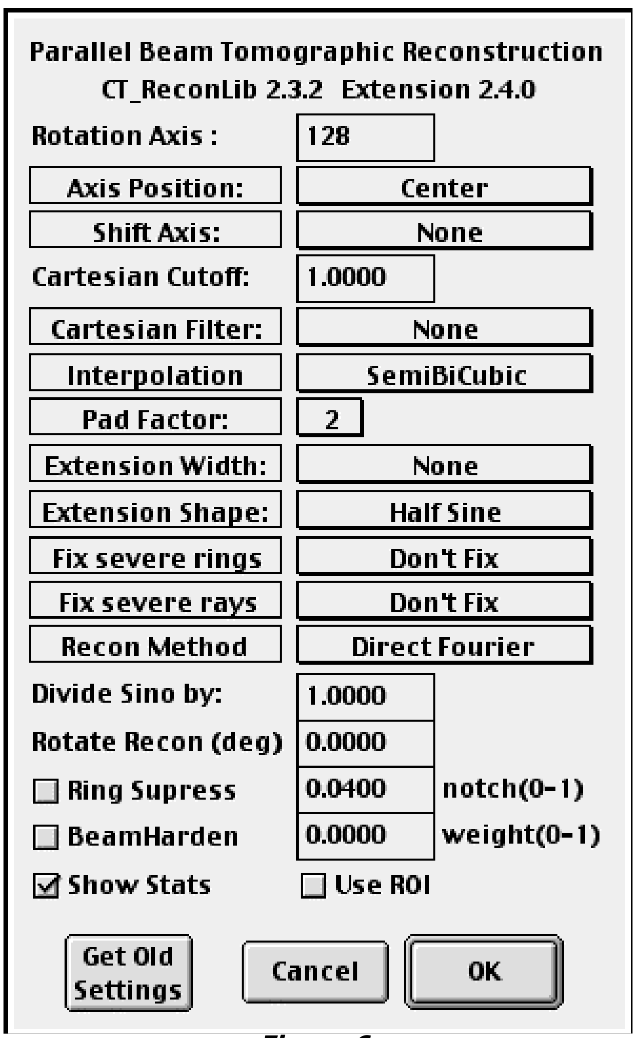

Figure 6

of the specimen. Analysis of these images to obtain image metrics relevant to the users research is, strictly speaking, beyond the purpose of the beamline. X2-B does provide a limited set of 3D analysis and visualization tools that can be made available to the user for use at the beamline. Further analysis can be done at the Center for Data Intensive Computing at BNL or at the users home facility using commercially available analysis, visualization and development packages such as Iris-Explorer, MatLab, AVS Express, IDL, PVWave, and 3dma(SUNY). The PRT does not endorse or support these packages but can supply file format specifications to aid in importing the images acquired at X2B.

Examples

In this example we acquire a 1024 cube of data of a sand pack and extract statistical properties of the pore space. The sand pack was prepared by pouring Ottawa sand, a common laboratory reagent, into a 1cm ID polyethylene tube. A 10μm/pixel pitch using the 2.5x lens was sufficient to image nearly the entire ID of the tube while capturing the pore shape and connectivity of this coarse-grained sand. The vertical dimension of the acquisition is limited by the beam height available at X2-B to about 5mm. If required the specimen height can be moved and 3D volumes concatenated to a vertical dimension of nearly 10cm. An x-ray beam energy of 25keV gave τ ~2 in the projection. The axis of rotation is at pixel column 512. The local protocol was used to acquire and reconstruct the projection data since the tube and a small portion of the

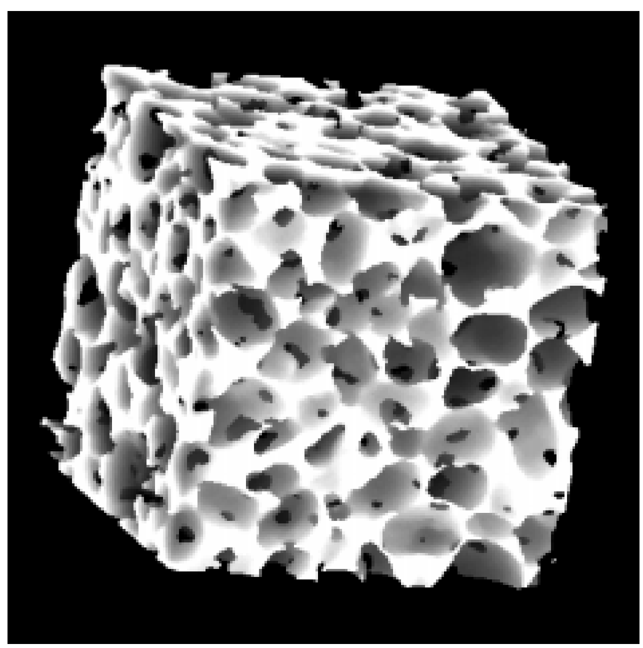

Figure 7

sand pack extended beyond the CCD field of view. A 256 cube was extracted from an arbitrary position within the 3D data and was visualized using VoxBlast, a commercial visualization package loaded on the reconstruction computer. The 256 cube was segmented into pore and solid using a simple grayscale threshold and rendered so that the solid sand grains are transparent revealing the 3D shape of the pore space as shown in **Figure 7**. 3DToolKit is a PRT extension to IPLab for analysis of 3D images. It contains tools for segmentation, connectivity, and statistical analysis of 3D images. In this example, we extract two statistical measures, the chord length and two point probability distributions from the full 3D data. The chord length distribution shown in **Figure 8** is the probability of finding a chord of length L in the 3D volume. The two-point probability function shown in **Figure 9** is the probability that two points selected randomly in the volume will both lie in this case in the pore phase. The asymptote of S(r) describes the correlation length for the pores, in this case about 250µm. These measures are related to transport, diffusion and trapping in random porous media.

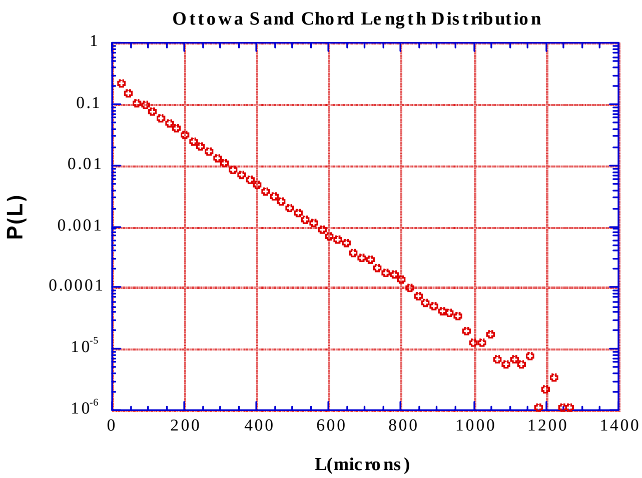

Figure 8

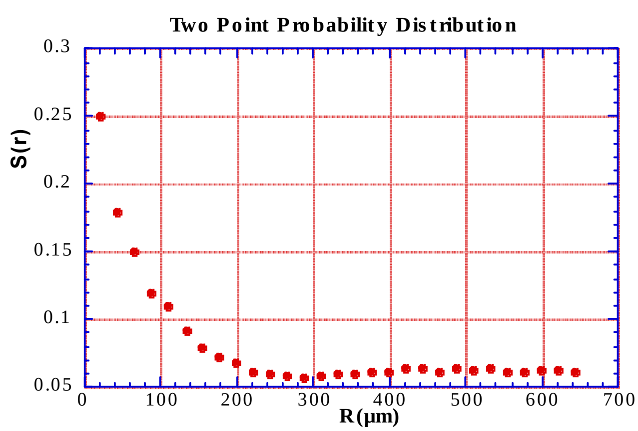

Figure 9

Useful Websites

IPLab
http://www.scanalytics.com/

CCD camera
http://www.roperscientific.com/

Iris Explorer
http://www.nag.co.uk/Welcome_IEC.html

AVS Express
http://www.avs.com/

Matlab
http://www.mathworks.com/

PVWave
http://www.vni.com/products/wave/index.html

VoxBlast
http://www.vaytek.com/

CDIC
http://www.bnl.gov/cdic/Sci_Projects/Computer_Sci/Visualization/visualization.htm

3DMA
http://www.ams.sunysb.edu/~lindquis/3dma/3dma.html

IDL
http://www.rsinc.com/idl/index.asp

CT Book
http://www.slaney.org/pct/

USEFUL REFERENCES

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"Principles of Computerized Tomographic Imaging", A.C. Kak and M. Slaney, IEEE press 1988 "Handbook of X-rays", E.F. Kaelble ed.,McGraw-Hill 1967.

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